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Department of Energy  
Carlsbad Field Office  
P. O. Box 3090  
Carlsbad, New Mexico 88221  
August 29, 2003



Mr. Steve Zappe, Project Leader  
Hazardous Waste Bureau  
New Mexico Environment Department  
2905 Rodeo Park Drive East, Bldg. 1  
Santa Fe, New Mexico 87505-6303

RE: Transmittal of the Final Audit Report for the Los Alamos National Laboratory  
Manual Headspace Gas Sampling (A-03-24)

Dear Mr. Zappe:

This letter transmits the Los Alamos National Laboratory (LANL) Audit Report for the processes performed to characterize and certify waste as required by Section II.C.2.c of the WIPP Hazardous Waste Facility Permit. The report contains the results of the audit performed. The audit was conducted July 8-10, 2003.

I certify under penalty of law that this document and all enclosures were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

Please contact the CBFO Quality Assurance Manager, Ava L. Holland, at (505) 234-7423 should you have any questions concerning this audit report.

Sincerely,

Dr. Ines R. Triay  
Manager

Enclosure



Mr. Steve Zappe

-2-

August 29, 2003

cc: w/o enclosure

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U.S. DEPARTMENT OF ENERGY  
CARLSBAD FIELD OFFICE

FINAL AUDIT REPORT

OF THE

LOS ALAMOS NATIONAL LABORATORY  
LOS ALAMOS, NEW MEXICO

WASTE CHARACTERIZATION ACTIVITIES  
HEADSPACE GAS SAMPLING AND ANALYSIS

AUDIT NUMBER A-03-24

July 8 – 10, 2003



Prepared By:

Thomas Putnam

Thomas Putnam, CTAC  
Audit Team Leader

Date:

8-26-03

Approved By:

Ava L. Holland

Ava L. Holland, CBFO  
Quality Assurance Manager

Date:

8/28/03

## 1.0 EXECUTIVE SUMMARY

Carlsbad Field Office (CBFO) Audit A-03-24 was conducted to evaluate the adequacy, implementation, and effectiveness of headspace gas (HSG) sampling, analysis, and associated activities utilizing the Entech/Agilent system.

The audit was conducted at the Los Alamos National Laboratory (LANL) in Los Alamos, New Mexico, July 8-10, 2003. The audit team concluded that the LANL process for obtaining manual HSG samples and analyzing those samples was adequate relative to the flow-down of requirements from the Hazardous Waste Facility Permit (HWFP). The audit team also concluded that the LANL technical processes were satisfactorily implemented and effective.

The audit team identified four isolated deficiencies requiring only remedial corrective actions that were corrected during the audit (CDA). Six recommendations were offered for management consideration.

## 2.0 SCOPE

The scope of the audit was to evaluate the LANL processes for adequacy, implementation, and effectiveness in performing sampling, analysis, canister cleaning, sample port installation, helium leak testing, batch data report generation, and the review of the batch data associated with the Entech/Agilent system. Compliance with the WIPP HWFP Waste Analysis Plan (WAP) and selected portions of the CBFO Quality Assurance Program Document (QAPD) was also evaluated.

The following Quality Assurance (QA) elements were evaluated:

- Personnel Qualification and Training
- Measuring and Test Equipment
- Sample Control

The following characterization technical elements were evaluated:

- HSG Sampling and Analysis
- Canister Cleaning
- Sample Port Installation
- Helium Leak Testing
- Generation Data Review
- Project-level Data Verification and Validation

The evaluation of LANL documents was based on the current revisions of the following documents:

- WIPP *Hazardous Waste Facility Permit*
- CBFO *Quality Assurance Program Document*, CAO-94-1012
- Related LANL technical and QA implementing procedures

### **3.0 AUDIT TEAM AND OBSERVERS**

#### **AUDITORS/TECHNICAL SPECIALISTS**

Thomas Putnam      Audit Team Leader, CTAC  
Dorothy Gill        Technical Specialist, CTAC

#### **INSPECTORS/OBSERVERS**

Steve Holmes        New Mexico Environment Department (NMED)  
Scott Webb           Environmental Evaluation Group (EEG)

### **4.0 AUDIT PARTICIPANTS**

LANL personnel participating in this audit process are identified in Attachment 1. A pre-audit meeting was held in the Oppenheimer Building on July 8, 2003. Daily meetings were held with LANL management and staff to discuss issues and potential deficiencies. The audit was concluded with a post-audit meeting held in the Oppenheimer Building on July 10, 2003.

### **5.0 SUMMARY OF AUDIT RESULTS**

#### **5.1 Program Adequacy, Implementation, and Effectiveness**

The audit team concluded that the LANL technical and QA processes/procedures were adequate relative to the flow-down of requirements from the CBFO QAPD and the HWFP. The audit team also concluded the LANL technical processes were satisfactorily implemented and effective.

The audit team concluded that the defined LANL QA program elements reviewed were adequate and satisfactorily implemented in accordance with the LANL Quality Assurance Program Manual (QAMP), the LANL Quality Assurance Project Plan (QAPjP), and LANL implementing procedures for the areas evaluated. The LANL QA program in those areas was also determined to be effective. For details of corrective action reports (CARs), CDAs, observations, recommendations, and exemplary practices, see Section 6.

A summary table of audit results for each of the QA program elements and the technical processes is provided in Attachment 2. Audit activities, including the specific objective evidence reviewed, are described below. A list of procedures evaluated during the audit is included in Attachment 3.

#### **5.2 Technical Activities**

Each technical area audited is discussed in detail in the following sections. The method used to select objective evidence is also discussed and the results of the assessments are provided. The objective evidence used to assess compliance with the WAP is cited and contained in Attachment 4.

### **5.2.1 Table B6-1 WAP Checklist**

The B6-1 WAP checklist addresses program requirements from a management perspective. It documents the verification that the waste characterization strategy, as defined in the WAP, is implemented by using controlled procedures. This audit was performed to assess LANL's ability to manually sample headspace gas using the new Entech/Agilent Headspace System. Objective evidence to evaluate the implementation of the associated characterization activities was selected and reviewed. Batch data reports, sampling records, measuring and test equipment calibrations, and training documentation for Transuranic (TRU) Waste Characterization Program (TWCP) personnel were included in the evaluation. The audit included direct observation of actual waste characterization activities (HSG sampling). Each characterization process involves:

- Collecting raw data
- Collecting quality assurance/quality control (QA/QC) information
- Reducing the data to a useable format, including a standard report
- Review of the report by the data generation facility and the site project office
- Comparing the data against program data quality objectives

The focus of the B6-1 checklist was to verify that LANL had implemented processes for the new HSG unit that covered areas from calibration of equipment through ensuring that headspace program data quality objectives were met. Items on the B6-1 checklist that are unaffected by the new sampling process are marked "NA."

During the audit, LANL demonstrated compliance with the characterization requirements of the WAP through documentation and by performing characterization activities. LANL provided documentation to support compliance with the WAP. Copies of these documents are included in Attachment 4. They include the reviewed batch data reports and measuring and test certifications.

### **5.2.2 Table B6-2 Solids and Soils/Gravel Sampling Checklist**

LANL is currently not certified to characterize homogeneous solid or soil/gravel waste streams. These processes were not audited during Audit A-03-24.

### **5.2.3 Table B6-3 Acceptable Knowledge Checklist**

The acceptable knowledge (AK) processes at LANL were not evaluated during Audit A-03-24. The AK processes have not changed as a result of the new Entech/Agilent Headspace System.

#### **5.2.4 Table B6-4 Headspace Gas Checklist**

Direct canister HSG sampling and associated activities were reviewed during the audit. The activities audited were documented in the following procedures:

- TWCP-DTP-1.2-069, *Installation of the NucFil HGAS Sample Port*
- TWCP-DTP-1.2-070, *Canister Cleaning Using Entech 3100 Canister Cleaning System*
- TWCP-DTP-1.2-071, *Manual Headspace Gas Sampling of LANL TRU Waste Containers*
- TWCP-DTP-1.2-072, *TRU Waste Container HGAS Analysis (Entech/Agilent)*
- TWCP-DTP-0.0-078, *Headspace GAS Sampling and Analysis Batch Data Reports Preparation (Entech/Agilent)*
- TWCP-DTP-0.0-079, *Entech Canister Gauge Leak Test*

Operators were knowledgeable with regard to their sampling duties, and the sampling processes were well organized. Batch data report generation and data validation processes were sufficiently comprehensive to meet all Waste Isolation Pilot Plant (WIPP) WAP requirements, and were well coordinated.

Successful installation of a sample port using TWCP-DTP-1.2-069, *Installation of the NucFil HGAS Sample Port*, was observed during the audit.

The areas of manual HSG sampling and sample port installation were determined to be adequate, satisfactorily implemented, and effective.

#### **5.2.5 B6-5 Radiography Checklist**

Radiography was not included in the scope of Audit A-03-24.

#### **5.2.6 B6-6 VE Checklist**

Visual examination was not included in the scope of Audit A-03-24.

### **6.0 CORRECTIVE ACTION REPORTS (CARs), CORRECTED DURING THE AUDIT (CDAs) OBSERVATIONS, RECOMMENDATIONS, AND EXEMPLARY PRACTICES**

During the audit, the audit team may identify conditions adverse to quality (CAQ) and document such conditions on CARs.

Condition Adverse to Quality (CAQ) – An all-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, nonconformances, and technical inadequacies.

Significant Condition Adverse to Quality – A condition which, if uncorrected, could have a serious effect on safety, operability, waste confinement, TRU waste site certification, compliance demonstration, or the effective implementation of the QA program.

## **6.1 Corrective Action Reports (CARs)**

No CARs were generated by the audit team during this audit.

## **6.2 Corrected During the Audit**

During the audit, the audit team may identify conditions adverse to quality (CAQ). The audit team members and the audit team leader (ATL) evaluate the CAQs to determine if they require a CAR. Once a determination is made that the CAQ does not require a CAR, the audit team members, in conjunction with the ATL, determine if the CAQ is an isolated case requiring only remedial action and, therefore, can be corrected during the audit (CDA). Upon determination that the CAQ is isolated, the audit team members, in conjunction with the ATL, evaluate/verify any objective evidence/actions submitted or taken by the audited organization and determine if the condition was corrected in an acceptable manner. Once it has been determined that the CAQ has been acceptably corrected, the ATL categorizes the condition as CDA.

Corrected During the Audit (CDA) – Isolated deficiencies that do not require a root cause determination or actions to preclude recurrence, and correction of the deficiency can be verified prior to the end of the audit. (Examples: one or two minor changes required to correct a procedure (isolated); one or two forms not signed or not dated (isolated); one or two individuals that have not completed a reading assignment.)

Four isolated deficiencies, requiring remedial action only, were identified during the audit and reported as CDAs. They were corrected and verified before the completion of the audit.

### **6.2.1 CDA 1**

Operators were not following procedure TWCP-DTP-1.2-069, *Installation of the Nucfil HGAS Sample Port*. During the checking of drum thickness, it was noted that the lid measured 0.0529 and the procedure required that the pounds per square inch (psi) be set at 90 to install the sample port. However, the operator stated that he rounds up when the reading is borderline (in this case, he rounded up to 0.0530). The procedure does not allow for rounding of the measured reading.



An interim change request was implemented that added the following text to Section 7.4.13:

"If the digital ultrasonic micrometer is a model that reads out more than three figures after the decimal place for the drum thickness listed in table 1, round the number in accordance with established rounding rules.

- If the digit 5, 6, 7, 8, or 9 is dropped, increase the preceding unit by 1 unit
- If the digit 0, 1, 2, 3, or 4 is dropped, do not alter the preceding digit"

#### **6.2.2 CDA 2**

Procedure TWCP-DTP-0.0-071, *Manual Headspace Gas Sampling of LANL TRU Waste Containers*, does not contain information on how the canisters are packaged for transportation to the laboratory, nor does it specify that the chain-of-custody form (COC) for canisters sampled is completed and placed in the shipping container to be taken to the laboratory.

An interim change request was implemented that added a new Section 8.7.5, as follows: "Samples are packed in appropriately cushioned and secured packaging so as to avoid canister damage during transport to the laboratory. For each sample batch, the COC Form will be packaged together with samples."

#### **6.2.3 CDA 3**

The temperature of the refrigerator being used to store volatile organic compound (VOC) liquid standards was measured at 1.3° centigrade (C) on July 1, 2003, and 0.8° C on July 9, 2003. Laboratory personnel stated that the standards should be stored at 4° C. Also, the storage requirement for liquid VOC standards is not specified in Procedure DTP-0.0-072.

An interim change request was implemented that added a second sentence to the bullet beginning "Custom liquid standard". "Standards will be stored at 4° C or in accordance with manufacturer's specifications." The manufacturer of the standards was contacted to ensure this was acceptable.

#### **6.2.4 CDA 4**

Procedure DTP-0.0-072 does not include all of the options used to select 4-bromofluorobenzene (BFB) scans used for tuning the mass spectrometer. SW-846 Method 8260B, Section 7.3.1.1, requires the laboratory to have a documented approach to scan selection.

An interim change request was implemented that replaced the Note at the end of Section 7.4.5.2.c with the following text. "Three methods of finding passing BFB may be used. First, check the spectrum at the apex of the BFB peak. Second, average three scans, the peak apex scan, and the scans immediately preceding and following the apex. Finally, average scans through the peak. These three methods may be used

with or without background subtractions. For background subtraction, use a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak."

### **6.3 Observations**

No observations were identified by the audit team during the audit.

### **6.4 Recommendations**

The following recommendations are provided for management consideration.

#### **6.4.1 Recommendation 1**

After the sampling event is completed, place a suitable trip blank in the shipping container with the samples.

#### **6.4.2 Recommendation 2**

The result sheet for the continuing calibration does not show the %D for hydrogen and methane. Hence it is not possible to determine from the sheet if the continuing calibration requirements were met. It is recommended that the sheet be revised to include this information.

#### **6.4.3 Recommendation 3**

SW 846 Method 8260B, Section 5.14, states that "all standards in methanol be stored at -10° C or less." The liquid VOC standards used by the laboratory are mixtures of pure compounds and are not dissolved in methanol. However, the standards contain methanol and it is recommended that the appropriateness of storing these standards at 4° C be investigated.

#### **6.4.4 Recommendation 4**

The results for hydrogen are reported to either 2 or 3 decimal places (e. g., batch LA03-HGAS/LA-001 hydrogen for drum 959196 was reported to 2 decimal places and that for drum 959150 was reported to 3 decimal places). A consistent reporting format should be applied.

#### **6.4.5 Recommendation 5**

The chromatograms for the quantitation reports have over-written analyte names that make them unreadable. It is recommended that the format be changed to ensure all analyte names are readable.

#### **6.4.6 Recommendation 6**

The Note on the "Gas Sample Chain of Custody" form from DTP-0.0-071 uses the words "sign and initial". However the samplers printed their names and initials. It is

recommended that either the form be changed to allow the printing of names or the samplers be instructed to sign their names.

## **7.0 LIST OF ATTACHMENTS**

Attachment 1: Personnel Contacted During the Audit

Attachment 2: Summary Table of Audit Results

Attachment 3: List of Procedures Audited

Attachment 4: Objective Evidence

**PERSONNEL CONTACTED DURING AUDIT A-03-07**

NAME	ORG/TITLE	PREAUDIT MEETING	CONTACTED DURING AUDIT	POST AUDIT MEETING
Anghel, Ioana	LANL/RRES-CH		X	X
Ankon, James	LANL/RRES-WDS		X	
Bailey, James	LANL/RRES-CE			X
Burt, Jean	LANL/RRES-QA		X	
Coriz, Suzanne	LANL/RRES-CH			X
Del Signore, J.C.	LANL/Project Manager	X		X
Fernandez, Ruby Ann	LANL/RRES-CE	X		X
Garcia, Mary Ann	LANL/RRES-CE	X		X
Gibson, Yvonne	LANL/RRES-CE	X		
Hardesty, Bill	LANL/HSG	X	X	X
Hartwell, Ware	LANL/RRES-QAT			X
Huchton, Judith	LANL/RRES-CE	X		X
Humphrey, Betty	LANL/SPM	X	X	X
Lindahl, Peter	LANL/SPQAO	X		X
Lopez, Joshua	LANL/RRES-WDS		X	
Marczak, Stanislaw	LANL/RRES-CH	X	X	X
Martin, Beverly	LANL/RRES-WD			X
Miller, Scott	LANL/RRES-CH	X		
Mullen, Richard	LANL/RRES-WDS		X	
Polley, Mark	RRES-AT/TCO			X
Powell, Mark	LANL/RRES-QAT		X	X
Newell, Dorothy	LASO-OPL			X
Nunz, James	LASO-OFO			X

NAME	ORG/TITLE	PREAUDIT MEETING	CONTACTED DURING AUDIT	POST AUDIT MEETING
Riggs, Matt	LANL/RRES-CE			X
Romero, Eric	LANL.RRES-WDS		X	
Saunders, Lori	LANL/QA			X
Sullivan, Jeri	LANL/RRES-CH		X	X
Uecker, Barbara	LANL/RRES-OEIM	X		X
Velasquez, Carmen	LANL/RRES-CE	X		X
Vigil, Chris	LANL/RRES-WDS		X	
Wander, Sandy	LANL/RRES-CE	X		X

Summary Table of Audit Results

Evaluation Area	Concern Classification			QA Evaluation			
	EP	CARs	CDAs	Obs	Rec	Adequacy	Implementation Effectiveness
Headspace Gas Sampling			2		4	A	S E
Sample Control			2		2	A	S E
Project Level V&V						A	S E
Training						A	S E
Measuring and Test Equipment						A	S E
<b>TOTALS</b>			<b>4</b>		<b>6</b>	<b>A</b>	<b>S E</b>

**Definitions**

E = Effective  
S = Satisfactory  
U = Unsatisfactory  
I = Indeterminate  
A= Adequate  
M= Marginal

CDA = Corrected During Audit  
CAR = Corrective Action Report  
Obs = Observation  
Rec = Recommendation  
EP=Exemplary Practice

## PROCEDURES AUDITED DURING A-03-24

NUMBER	PROCEDURE NUMBER	TITLE
1.	QP-1.1-003	TWCP Training
2.	QP-1.1-010	Project Level Data Validation and Verification
3.	QP-1.1-018	Measuring and Test Equipment
4.	DTP-1.2-069	Installation of the NucFil HGAS Sample Port
5.	DTP-00-070	Canister Cleaning Using Entech 3100 Canister Cleaning System
6.	DTP-00-071	Manual Headspace Gas Sampling of LANL TRU Waste Containers
7.	DTP-00-072	TRU Waste Container HGAS Analysis (Entech/Agilent)
8.	DTP-00-078	Headspace Gas Sampling and Analysis Batch Data Reports Preparation (Entech/Agilent)
9.	DTP-00-079	Entech Canister Gauge Leak Test